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TECHNOLOGIES FOR TESTING FUEL QUALITY

Approaches for the Petroleum Quality Analysis System (PQAS)

INTERIM REPORT
TFLRF No. 345

by

S.R. Westbrook
U.S. Army TARDEC Fuels and Lubricants Research Facility (SwRI)
Southwest Research Institute
San Antonio, TX

Under Contract to

U.S. Army TARDEC
Petroleum and Water Business Area
Warren, MI 48397-5000

Contract No. DAAK70-92-C-0059

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Approved by:

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13. ABSTRACT (Maximum 200 words)

The Petroleum Quality Analysis System (PQAS) was demonstrated in a recently completed PDRR (Program Definition and Risk Reduction) phase. The PDRR phase demonstrated several new technologies for testing fuel quality. Over 20 fuel-quality properties were analyzed using these technologies. Since that time, CASCOM (Combined Arms Support Command) has shortened the list of required tests. This shift in attitude toward performance requirements signals a new responsibility for TARDEC to further refine the list of required tests and consider future requirements.

Under this program, the TARDEC Fuels and Lubricants Research Facility (TFLRF) at Southwest Research Institute (SwRI) in San Antonio, Tx, investigated solutions to poor correlation on a number of properties reported in Interim Report TFLRF No. 321, "Estimation of Middle Distillate Fuel Properties by FT-IR and Chemometrics: Part I - Calibrations and Validations." The essential fuel properties necessary to determine fuel quality in the field were also identified. Specific tasks included the following: 1) identify those kerosene fuel properties deemed essential or highly beneficial in indicating fuel quality and usability in military ground vehicle engines; 2) evaluate whether infrared techniques, particularly FT-IR, can be used as a measuring tool to determine any or all of the fuel-quality properties identified in the above task; and 3)identify and recommend a combination of instruments capable of adequately characterizing fuel performance, quality, or usability, and determine which best satisfies the PQAS requirements.

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PQAS FT-IR Fuel Analysis Infrared Spectroscopy Fuel Test Methods

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EXECUTIVE SUMMARY

Problems and Objectives: The Petroleum Quality Analysis System (PQAS) was demonstrated in a recently completed PDRR (Program Definition and Risk Reduction) phase. The PDRR phase demonstrated several new technologies for testing fuel quality. Over 20 fuel-quality properties were analyzed using these technologies. Since that time, CASCOM (Combined Arms Support Command) has shortened the list of required tests. This shift in attitude toward performance requirements signals a new responsibility for TARDEC to further refine the list of required tests and consider future requirements.

Importance of Project: The U.S. Army CASCOM and Quarter Master School placed one primary requirement on the PQAS; it must meet the testing protocols specified in MIL-HDBK-200G. This is necessary because the U.S. Army must be able to support operations by its allies, and all test data must be recognizable by all parties involved. Nonstandard test methods may produce results that are of little or no value to U.S. troops or allies. In short, this means that test methods must be ASTM standard methods or their equivalent. Any correlative or estimated methods must produce results that are equivalent to the standard method.

Technical Approach: Under this program Southwest Research Institute identified the essential fuel properties necessary to determine fuel quality in the field and investigated solutions to poor correlation between standard and nonstandard test method results for a number of the identified properties.

Specific tasks included the following:

- Identify those kerosene fuel properties deemed essential or highly beneficial in indicating fuel quality and usability in military ground vehicle engines.
- Evaluate whether infrared techniques, particularly FT-IR, can be used as a measuring tool to determine any or all of the fuel-quality properties identified in the above task.
- Identify and recommend a combination of instruments capable of adequately characterizing fuel performance, quality, or usability, and determine which best satisfies the PQAS requirements.

Accomplishments: A list of recommended test methods was compiled and justified.

Military Impact: The use of the test methods identified in this report will allow Army personnel to more quickly measure the quality of fuel and reduce the chance of using contaminated or unacceptable fuel.

FOREWORD/ACKNOWLEDGMENTS

This work was performed by the U.S. Army TARDEC Fuels and Lubricants Research Facility (TFLRF) located at Southwest Research Institute (SwRI), San Antonio, Texas, during the period October 1998 through March 1999 under Contract No. DAAK70-92-C-0059. The work was funded and administered by the U.S. Army Tank-Automotive RD&E Center, Petroleum and Water Business Area, Warren, Michigan. Funding was administered by Mr. Luis Villahermosa (AMSTA TR-D/210), who served as the TARDEC contracting officer's representative and project technical monitor.

The author would like to recognize Andrew Schutt and Steve Moyer for their assistance, guidance, and review of this report.

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ACRONYMS & ABBREVIATIONS

TACOM	U.S. Army Tank-automotive Armaments Command
TARDEC	U.S. Army Tank Automotive Research, Development and Engineering Center
TFLRF	U.S. Army TARDEC Fuels and Lubricants Research Facility
SwRI	Southwest Research Institute
CASCOM	Combined Arms Support Command
ASTM	American Society for Testing and Materials
FT-IR	Fourier Transform Infrared Spectroscopy
PQAS	Petroleum Quality Analysis System

APPLICABLE DOCUMENTS

Government Documents

Specifications, Standards, and Handbooks. The following specifications, standards, and handbooks form a part of this document to the extent specified herein. Unless otherwise specified, the latest issues of these documents are those listed in the issue of the Department of Defense Index of Specifications and Standards (DoDISS).

Federal specifications

A-A-52530, Gasohol, Automotive, Unleaded
A-A-52557, Fuel Oil, Diesel, For Post Camp and Station

DoD Specifications

MIL-PRF-5624, Turbine Fuel, Aviation, Grades JP-4, JP-5, and JP-5/JP-8 ST
MIL-T-83133, Turbine Fuels, Aviation, Kerosene Types, NATO F-34 (JP-8) and NATO F-35
MIL-F-16844, fuel, Naval distillate, NATO F-76
MIL-I-25017, Corrosion/Lubricity Improver, Fuel Soluble (Metric)
MIL-I-27686, Inhibitor, Icing, Fuel System, NATO S-748
MIL-I-85470, Inhibitor, Icing, Fuel system, High Flash, NATO Code Number S-1745
MIL-S-53021, Stabilizer Additive, Diesel Fuel

Federal Standards

ISO 10012, Part I, International Standards Organization, Quality Assurance Requirements for Measuring Equipment, Meteorological Confirmation system for Measuring Equipment.
OSHA STDS 29 CFR 1910, OSHA Safety and health Standards.
Federal Test Method Standard No. 791 - Lubricants, Liquid Fuels, and Related Products; Methods of Testing

ASTM STANDARDS

D 86	Distillation of Petroleum Products
D 93	Flash Point by Pensky-Martens closed Tester.
D 97	Pour Point of Petroleum Oils.
D 156	Saybolt Color of Petroleum Products.
D 445	Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic viscosity).
D 613	Ignition Quality of Diesel Fuels by the Cetane Method.
D 975	Diesel Fuel Oils.
D 976	Calculated Cetane Index of Distillate Fuels.
D 1094	Water Reaction of Aviation Fuels
D 1298	Density, Relative Density, or API Gravity of Crude Petroleum and Liquid Hydrocarbon Products by Hydrometer Method.
D 1319	Hydrocarbon Types in Liquid Petroleum Products by Fluorescent Indicator Adsorption.
D 1368	Trace Concentrations of Lead in Primary Reference Fuels.
D 1500	ASTM Color of Petroleum Products (ASTM Color Scale)
D 1655	Aviation Turbine Fuels.
D 2276	Particulate Contamination in Aviation Fuel by Line Sampling.
D 2386	Freezing Point of Aviation Fuels.
D 2500	Cloud Point of Petroleum Products.
D 2622	Sulfur in Petroleum Products (X-Ray Spectrographic Method).
D 2624	Electrical Conductivity of Aviation and Distillate Fuels Containing Static Dissipator Additive.
D 2699	Knock Characteristics of Motor Fuels by the Research Method.
D 2700	Knock Characteristics of Motor and Aviation Fuels by the Motor Method.
D 2885	Research and Motor Method Octane Ratings using On-line Analyzers.
D 3116	Trace Amounts of Lead in Gasoline.
D 3348	Trace Lead in Unleaded Gasoline (Colorimetric Method)
D 3948	Method for Determining Water-Separation Characteristics of Aviation Turbine Fuel by Portable Separometer.
D 4052	Density and Relative Density of Liquids by Digital Density Meter.
D 4176-93 (1997)	Standard Test Method for Free Water and Particulate Contamination in Distillate Fuels (Visual Inspection Procedures)
D 4294	Sulfur in Petroleum Products by Energy-Dispersive X-Ray Fluorescence Spectroscopy.
D 4308	Electrical Conductivity of Liquid Hydrocarbons by Precision Meter.
D 4737	Calculated Cetane Index by Four Variable Equation.
D 4814	Automotive Spark-Ignition Engine Fuel.
D 4815	Determination of C1 to C4 Alcohols and MTBE in Gasoline by Gas Chromatography.
D 5006	Measurement of Fuel System Icing Inhibitors (Ether Type) in Aviation Fuels.
D 5059	Lead in Gasoline by X-Ray Spectroscopy.
D 5190	Vapor Pressure of Petroleum Products (Mini Method)
D 5452	Particulate Contamination in Aviation Fuels by Laboratory Filtration.
D 5972	Freezing Point of Aviation Fuels (Automatic Phase Transition Method)
D 6045	Color of Petroleum Products by the Automatic Tristimulus Method
D 6426-99	Standard Test Method for Determining Filterability of Distillate Fuel Oils
D 6450	Flash Point by Continuously Closed Cup

I. INTRODUCTION AND BACKGROUND

The Petroleum Quality Analysis System (PQAS) was demonstrated in a recently completed PDRR (Program Definition and Risk Reduction) phase that demonstrated several new technologies for testing fuel quality. Over 20 fuel-quality properties were analyzed using these technologies. Since that time, CASCOM (Combined Arms Support Command) has shortened the list of required tests. This shift in attitude toward performance requirements signals a new responsibility for TARDEC to further refine the list of required tests and consider future requirements.

II. APPROACH

Under this program, the TARDEC Fuels and Lubricants Research Facility (TFLRF) at Southwest Research Institute (SwRI) in San Antonio, TX, investigated solutions to poor correlation on a number of properties reported in Interim Report TFLRF No. 321, "Estimation of Middle Distillate Fuel Properties by FT-IR and Chemometrics: Part I – Calibrations and Validations." The essential fuel properties necessary to determine fuel quality in the field were also identified. Specific tasks included the following:

- Identify those kerosene fuel properties deemed essential or highly beneficial in indicating fuel quality and usability in military ground vehicle engines.
- Evaluate whether infrared techniques, particularly FT-IR, can be used as a measuring tool to determine any or all of the fuel-quality properties identified in the above task.
- Identify and recommend a combination of instruments capable of adequately characterizing fuel performance, quality, or usability, and determine which best satisfies the PQAS requirements.

III. PQAS REQUIREMENTS

The U.S. Army CASCOM and Quarter Master School placed one primary requirement on the PQAS; it must meet the testing protocols specified in MIL-HDBK-200G. This is necessary because the U.S. Army must be able to support operations by its allies, and all test data must be recognizable by all parties involved. Nonstandard test methods may produce results that are of little or no value to U.S. troops or allies. In short, this means that test methods must be ASTM standard methods or their equivalent. Any correlative or estimated methods must produce results equivalent to the standard method.

In addition to the above requirement, the PQAS has size, power, and analysis time requirements.

IV. FUEL TESTING REQUIREMENTS

The protocols and requirements for fuels owned by the Army are detailed in MIL-HDBK-200G, dated 1 July 1987. The specific properties to measure and the frequency of analysis are dependent on the type or grade of fuel and the intended use of the fuel. For each product, the levels of testing (i.e. the extent of testing) are divided into levels: A, B-1, B-2, B-3, and C. The goal for the PQAS is to provide the capability to perform as many B-3 level diesel and aviation-kerosene fuel tests as possible. According to MIL-HDBK-200G, tests at the B-3 level are "a partial analysis for contamination; particularly for controlling the reinjection of pipeline interface products." Table 1 lists the tests required for B-3 testing of all fuels used by the Army.

Recognizing that some of these tests are difficult to perform under the constraints of the PQAS, a secondary goal was established. This goal was for the PQAS to provide the capabilities currently present in the Army's Airmobile Laboratory.(1)* Table 2 lists the B-3 requirements (for all fuel types) as well as the tests included in the Airmobile Laboratory. The list shows that the Airmobile Laboratory is capable of performing the following: visual appearance, density, solids, distillation, copper strip corrosion, Reid vapor pressure, flash point, water reaction, fuel system icing inhibitor, and filtration time. The list of tests in the Airmobile laboratory is shorter. However, some of those test methods are still difficult to perform under the conditions/constraints of the PQAS. For example, the copper strip corrosion test is difficult to include in the PQAS because it requires the means to heat the fuel to 100°C for 3 hours. This is a safety hazard and a significant power requirement. Particulate contamination is also difficult because it requires a drying oven, a vacuum source, and volatile solvents.

*Underscored numbers in parentheses indicate references listed at the end of the document.

Table 1. Requirements For Type B-3 Testing

Test Requirements	Aviation Turbine	Diesel and Kerosene	Aviation Gasoline	Motor Gasoline	Notes
Water and Solids	X	X	X	X	
Visual Color	X		X	X	According to the test method: "This method is used for determining the acceptability of color of dyed gasolines (aviation or motor)." This method should not be used for aviation turbine fuel; the Saybolt color test should be used for aviation turbine fuels.
Density	X	X	X	X	Digital density meter.
Solids (Millipore)	X		X		• ASTM D 5452, Appendix X1. • AEL MK III or MK IV; see Appendix G of MIL-HDBK-200G. This would be applicable to aviation turbine but not aviation gasoline. • ASTM D 2068 for diesel fuel.
Distillation	X		X	X	
Copper Strip Corrosion	X		X	X	
Freezing Point	X				
Existent Gum	X				
Reid Vapor Pressure	X				(JP-4 only)
Flash Point	X	X			(Except JP-4)
Water Reaction	X		X		
Lead Content	X			X	(If contamination with leaded fuels is suspected)
Fuel System Icing Inhibitor	X				
Filtration Time	X				
Water Separation Index	X				
Conductivity	X				
Unwashed Gum				X	
Water Tolerance				X	Gasohol Only
Lean Mixture Rating			X		If the capability does not exist to perform this test at the terminal, a sample will be sent to the nearest service laboratory that does have the capability. In the event operational necessity dictates issue of product before results are obtained from the service laboratory, shipments may be made. However when laboratory results indicate failure on a recurring basis, notify DESC.
Color		X			

Table 2. B-3 Requirements vs. Airmobile Laboratory Capabilities

	Test Requirements	Aviation Turbine	Diesel and Kerosene	Aviation Gasoline	Motor Gasoline	In Airmobile Laboratory	Notes
1	Water and Solids	x	x	x	x	y	Uses 1 quart glass jar or beaker. The white bucket is used to detect the presence of red dye in aviation turbine fuel.
2	Visual color	x		x	x	n	According to the test method: "This method is used for determining the acceptability of color of dyed gasolines (aviation or motor)." This method should not be used for aviation turbine fuel; the Saybolt color test should be used for aviation turbine fuels.
3	Density	x	x	x	x	y	Recommend using density meter rather than hydrometers and glass cylinders.
4	Solids (Millipore)	x		x	x	y	Recommend using AEL MK III or MK IV; see Appendix G of MIL-HDBK-200G. This would be applicable to aviation turbine but not aviation gasoline.
5	Distillation	x		x	x	y	A gas chromatograph could be used but would not be as sensitive to high-boiling point contaminants.
6	Copper Strip Corrosion	x		x	x	y	Will require a heating bath or electric heating block.
7	Freezing Point	x					Would require source of dry ice or use of Phase Technology Automatic tester.
8	Existent Gum	x				n	Should be dropped from the PQAS.
9	Reid Vapor Pressure	x				y	(JP-4 only)
10	Flash Point	x	x			y	(Except JP-4) Recommend using the Grabner Automatic Tester.
11	Water Reaction	x		x		y	Requires concentrated sulfuric acid solution to clean the glassware.
12	Lead Content	x			x	n	(If contamination with leaded fuels is suspected)
13	Fuel System Icing Inhibitor	x				y	Use the B-2 test kit.
14	Filtration Time	x				y	
15	Water Separation Index	x				n	Microseparometer
16	Conductivity	x				n	Handheld conductivity meter
17	Unwashed Gum				x	n	See existent gum above
18	Water Tolerance				x	n	Gasohol Only
19	Lean Mixture Rating			x		n	If this test can't be performed at the terminal, a sample will be sent to the nearest service laboratory that does have the capability. In the event operational necessity dictates issue of product before results are obtained from the service laboratory, shipments may be made. However, when laboratory results indicate failure on a recurring basis, notify DESC.
20	Color		x			n	D1500 color-Recommend Minolta automatic tester to get D156 and D1500

One option for meeting the PQAS requirements is to use alternative or nontraditional methods. In order to meet Army requirements, an alternative method must produce results equivalent to the specified ASTM method. It is preferable that the alternative method be ASTM approved and recognized by ASTM as an alternative. Another option, though less preferable, is that the alternative method be ASTM approved even if it is not currently recognized as an allowed alternative method of analysis for that property.

The purpose of this project was to investigate test-method options that meet the requirements of the PQAS that are not met by the currently specified ASTM methods. In some cases, a suitable alternative method exists. In some cases, an alternative exists that is not ASTM-approved. In still others, no suitable alternative exists, and the PQAS requirements need to be revised.

V. TEST METHODS

Each B-3 test is discussed below. If the current method meets PQAS requirements, no further discussion is given; it is assumed that the Army will continue to include that test in the PQAS. If necessary, alternative methods are presented along with a discussion of the significance of the test results. The discussion also includes any available information on the correlation of test results. If no alternative exists, the implications of keeping the test in the PQAS is discussed. Table 3 summarizes this section.

A. (1)* Visual Appearance (Water and Solids) – This is a visual examination of the fuel and meets the PQAS requirements.

B. (2) Visual Color – The specified test method is Method 103 of FTMS 791. This tests for the color of dyed gasoline and is rarely used. There is currently no automatic method. The standard, manual, visual color comparator, which meets the PQAS requirements, could be used. Since this test is outdated and rarely called for, the Army should consider dropping it from the PQAS.

*Bolded numbers in parentheses indicate corresponding item number in Table 2.

C. (3) Density – Using a meter that meets the requirements of ASTM D 4052, Standard Test Method for Density and Relative Density of Liquids by Digital Density Meter, is recommended. A meter with a computer interface is preferred but not required.

D. (4) Solids—This is a test for particulate contamination in the fuel. The specified test methods for solids are D 2276, D 5452, or Appendix A, MIL-PRF-5624. The specified procedure involves passing the fuel through two, pre-weighed filters. The filters are then solvent rinsed, oven dried, and re-weighed. Performing this test requires a vacuum source, hazardous solvents, an analytical balance, and a drying oven. All of these are difficult in the currently envisioned PQAS due primarily to size and power requirements.

It is important to note that this test is included in the Airmobile Laboratory, but it is a B-3 requirement only for aviation fuel. However, fuel handlers also use the test to measure the effectiveness of fuel filters on fuel-handling equipment. Under this second application, the test could be used with diesel fuel. It is also useful to measure the cleanliness of diesel fuel although this is not a B-3 requirement. The diesel fuel applications are mentioned because they are useful; however, they do complicate finding an alternative to the standard gravimetric procedure.

Three alternatives are available for the aviation kerosene application; each is discussed below:

1. The first alternative involves filtering the fuel through the same filters used in the gravimetric method. The filters are then visually compared to standards published by ASTM. This method is detailed in ASTM D 5452 and Appendix X1 to ASTM D 5452. The method describes how to determine the filter membrane color rating of an aviation fuel sample. The filter membrane color rating may be used for the qualitative assessment of contaminant levels in the fuel based on the color of the material on the filter. Note that this is a qualitative procedure; it could be used as a pass/fail method. Failed fuels could then be submitted for gravimetric analysis at an appropriate laboratory. This method is not applicable to diesel fuel because diesel fuel can stain the test filters without adding weight or plugging the filter.

2. The second alternative is similar to the first. It involves using the AEL-MKIII Contaminated Fuel Detector described in MIL-HDBK-200G. The fuel is filtered through a filter membrane as with the gravimetric procedure. The filter is then placed in a photocell assembly. A light is shined on the filter, and the amount of light that passes through the filter is measured. The light reading is converted to an equivalent weight of particulates using a calibration curve. Again, this method is not applicable to diesel fuel for the same reason mentioned above.

3. The third alternative is ASTM D 6426, Standard Test Method for Determining Filterability of Distillate Fuel Oils. In this test, a sample of the fuel to be tested is passed at a constant flow rate through a glass fiber filter. The pressure drop across the filter is monitored during the passage of a fixed volume of test fuel. If a prescribed maximum pressure drop is reached before the total volume of fuel is filtered, the actual volume of fuel filtered at the time of maximum pressure drop is recorded. This is also a pass/fail test with specific requirements set by the user. This test method is applicable to diesel fuel but is not a recognized method for aviation fuel cleanliness. At the time of this writing, ASTM is working toward an equivalent method for aviation turbine fuel.

It is recommended that one of the two following approaches be used for this test:

- Equip the PQAS to test aviation kerosene only. Use alternative 1 or 2 above.
- Equip the PQAS to test both aviation and diesel fuel. Use alternative 1 or 2 for aviation fuel. Use alternative 3 for diesel fuel.

E. (5) Distillation—The specification-required test method calls for the controlled distillation of 100 milliliters of the sample fuel. The apparatus typically used for this test consumes large amounts of power. It also requires water and air and covers about 3 to 4 cubic feet of space. While it is possible to perform this test in the PQAS, it would be very difficult under the current requirements. A potentially more acceptable approach is to determine distillation by ASTM D 2887. This is a gas chromatographic method. Separation Systems, Inc. has developed a system that performs this test in about 7 to 10 minutes. The system involves a special injector and changes to the heating rate. Some work would be required to select the proper gas chromatograph for the PQAS requirements. In addition, D 86 and D 2887 provide different results. However, the correlation between the two is highest in the middle of the distillation curve (the area of greatest interest); ASTM is working on a correction factor.

F. (6) Copper Strip Corrosion—This test involves placing a polished copper strip in a measured volume of the test fuel and heating the fuel for 2 to 3 hours based on the type of fuel. The copper corrosion test is primarily used to test for the presence of corrosive sulfur. It also shows the presence of some halogenated compounds. This method allows two means of heating the sample, an oil bath or a heated metal block. While testing in the PQAS could be accomplished with a metal heating block, the time requirements of the standard test would exceed the currently expected PQAS constraints. There is no viable alternative for this test. Since this is primarily a test for corrosive sulphur, it could be argued that fuels with very low levels of sulfur, such as current low-sulfur fuels, pose only a small risk of failing the test. The Army is currently funding a study of this approach. For the purposes of the PQAS, a sulfur level could be set so that the copper strip corrosion test is not required. When the level of sulfur in the fuel exceeds the given level, the fuel could be used with caution or sent to a laboratory for copper corrosion testing.

It is recommended that the maximum sulfur level approach be considered and that this method be dropped from the list of tests in the PQAS.

G. (7) Freezing Point—It is recommended that an automatic tester meeting the requirements of ASTM D 5972, Standard Test Method for Freezing Point of Aviation Fuels (Automatic Phase Transition Method), be used to measure the freezing point. A tester that operates without added cooling should be used to reduce the needed space and logistical support.

H. (8) Existent Gum—This test is used to determine the amount of very high molecular weight compounds such as gums and some additives in gasoline and aviation fuels. The test involves evaporating the fuel with a jet of superheated air or steam. The residue is then weighed to determine the result. This test requires an analytical balance, glass beakers, acid for cleaning the beakers, a large source of compressed air, a steam generator, a superheater, and a heated metal evaporation block. This method does not meet the power and size requirements of the PQAS. It is also an extreme health and safety risk because of the superheated air/steam and the acid cleaning solution. There is currently no viable alternative for this test. This test is used at the B-3 level mainly to detect contamination. Other tests, such as color and distillation, could probably adequately serve that purpose.

It is recommended that this test be dropped from the PQAS.

I. (9) Reid Vapor Pressure—This test is for gasoline and JP-4. It is recommended that an automatic tester conforming to the requirements of ASTM D 5190, Standard Test Method for Vapor Pressure of Petroleum Products (Mini Method), be used for this test. Such an instrument meets the requirements of the PQAS.

J. (10) Flash Point – It is recommended that an instrument conforming to ASTM D 6450, Standard Test Method for Flash Point by Continuously Closed Cup, be used to measure flash point in the PQAS.

K. (11) Water Reaction—This is a manual test to evaluate the emulsion-forming tendencies of a fuel. A sample of the fuel is shaken, using a standardized technique, at room temperature with a phosphate buffer solution in scrupulously cleaned glassware. The change in volume of the aqueous layer and the appearance of the interface are taken as the water reaction of the fuel.

The only potential problem with this test is the need for scrupulously cleaned glassware. A chromic acid solution is the method's prescribed cleaning agent. Since this cleaning solution presents a severe safety hazard, it should not be carried in the PQAS. It is recommended that the microseparometer (see item O below) be used to evaluate the emulsion-forming tendencies in routine use. When the water-reaction test is needed, pre-cleaned glassware and containers of buffer solution could be used. The glassware would be good for only one analysis and would then be disposed or sent for recleaning and packaging. Since storage space is limited, it is likely that only a small number of water reaction test kits could be carried in the PQAS.

L. (12) Lead Content – This test is applicable to motor and aviation gasoline, which are a relatively low priority for the PQAS. It is also used with aviation turbine fuel if contamination with leaded aviation gasoline is suspected. If it is necessary to know the amount of lead in a gasoline sample, the fuel could first be screened using ASTM D 3348, Standard Test Method for Trace Lead in Unleaded

Gasoline (Colorimetric Method). This is a field method for the quantitative measurement of lead in unleaded gasoline ranging from 0.01 to 0.10 g Pb/U.S. gal (2.64 to 26.4 mg Pb/L). If the level of lead in the sample exceeds 26.4 mg/L, the fuel should then be tested using ASTM D 5059, Standard Test Method for Lead in Gasoline by X-ray Spectroscopy.

A suitable X-ray spectrometer could be included in the PQAS if space permits. However, it is recommended that only ASTM D 3348 be included and that any fuel exceeding the method's maximum detection limit be considered a leaded gasoline and used appropriately. Keeping a spectrometer tuned and calibrated for rare occasions requiring lead analysis is an unwarranted use of valuable resources.

M. (13) Fuel System Icing Inhibitor—It is recommended that the standard B-2 test kit for fuel system icing inhibitor be used.

N. (14) Filtration Time— The specified test method is found in Appendix A of MIL-PRF-5624. It will be difficult to run this test (as prescribed by the method) under the requirements of the PQAS. The standard test involves measuring the time required to filter one gallon of fuel. This requires a large filter flask and a vacuum pump. A specially constructed vacuum chamber, sized to hold 1.5 gallons of fuel and made of steel or aluminum, could be built into one of the workbenches in the PQAS. A vacuum pump, connected to the vacuum chamber, would also be required. The vacuum chamber would need a top opening, for the filter funnel, and a drain.

A modified filtration time test could be developed using the AEL MK-III. However, this is not recommended since it would not meet the ASTM requirements.

O. (15) Water Separation Index—Use ASTM D 3948, Standard Test Method for Determining Water Separation Characteristics of Aviation Turbine Fuels by Portable Separometer.

P. (16) Conductivity—Use ASTM D 2624, Standard Test Methods for Electrical Conductivity of Aviation and Distillate Fuels.

Q. (17) Unwashed Gum— This test uses the same equipment used for existent gum (item H above). The same reasoning used for existent gum above, applies here. It is recommended that this test be dropped from the PQAS.

R. (18) Water Tolerance—This test is designed to estimate the tolerance of gasoline/alcohol blends to the presence of water. Since this is a test for a low-priority, rarely-tested product, it is recommended that this test be dropped from the PQAS.

S. (19) Lean Mixture Rating—This is an engine test to estimate the ignition quality of aviation gasoline. There is no way to run the test in the PQAS, and there is no ASTM alternative. For these reasons, and since this is a test for aviation gasoline, it is recommended that the test be dropped from the PQAS.

T. (20) Color— This is ASTM D 1500, Standard Test Method for ASTM Color of Petroleum Products (ASTM Color Scale). There are both manual and automatic testers that produce D 1500 results. ASTM D 6045, Standard Test Method for Color of Petroleum Products by the Automatic Tristimulus Method, uses an automatic tester that measures the ASTM color of fuels and is also capable of measuring the Saybolt color (ASTM D 156) of aviation fuels. It is recommended that the PQAS include the instrument used for D 6045.

Table 3 summarizes this section of the report.

Table 3. Summary of Tests and Test Equipment for PQAS				
	Test Requirements	**In Airmobile Laboratory**	**Include in PQAS**	**Notes**
1	Water and Solids	y	y	1 quart jar and white bucket
2	Visual color	n	n	Manual color comparator if test is needed. Consider dropping the test from the PQAS
3	Density	y	y	ASTM D4052
4	Solids (Millipore)	y	y	•ASTM D5452, Appendix X1 •AEL MK III or MK IV; see Appendix G of MIL-HDBK-200G. This would be applicable to aviation turbine but not aviation gasoline •ASTM D2068 for diesel fuel
5	Distillation	y	y	Automatic distillation appratus or ASTM D2887
6	Copper Strip Corrosion	y	n	Recommend the test be dropped. If required, an electric heating bath, with a stainless steel enclosure for test tube, could be used
7	Freezing Point	n	y	ASTM D5972
8	Existent Gum	n	n	Recommend the test be dropped
9	Reid Vapor Pressure	y	y	ASTM D5190
10	Flash Point	y	y	ASTM D6450
11	Water Reaction	y	y	Use pre-packaged glassware and buffer. Use once, then dispose or recycle
12	Lead Content	n	n	ASTM D3348 as a screening tool
13	Fuel System Icing Inhibitor	y	y	B-2 test kit.
14	Filtration Time	y	y	AEL MK-III
15	Water Separation Index	n	y	ASTM D3948
16	Conductivity	n	y	ASTM D2624
17	Unwashed Gum	n	n	Recommend the test be dropped
18	Water Tolerance	n	n	Recommend the test be dropped
19	Lean Mixture Rating	n	n	Recommend the test be dropped
20	Color	n	y	ASTM D6045

VI. INFRARED ANALYSIS METHODS

The use of indirect methods that utilize techniques such as infrared spectroscopy is increasing. These methods rely on a correlation between some property such as infrared absorptions and fuel properties such as cetane number or heat of combustion. Indirect methods are not as accurate as direct measurements. Table 4 lists some gasoline and diesel fuel properties that can be estimated by infrared spectroscopy. Table 5 provides representative correlation coefficients (ASTM method vs. infrared) for selected fuel properties. These correlation coefficients are only representative of the results that can be obtained. The correlation of any given calibration model is dependent on several factors including number of calibration fuels, types of fuels, statistical protocol used, quality of reference laboratory data, and the infrared spectrometer used. Table 6 presents representative standard error of prediction (SEP) values for selected diesel/kerosene properties. As with the correlation coefficients, SEP values are dependent on several factors.

The primary advantages of infrared methods are rapid analysis time and the ability to estimate several fuel properties from a single infrared scan. Currently, there are no standardized ASTM methods for the indirect measurement of fuel properties using infrared spectroscopy. There is a standardized infrared method for determining benzene in gasoline; ASTM is working on a standard practice for quantitative determination of hydrocarbon types in spark-ignition engine fuels by infrared spectroscopy. There is no current work on methods or practices for other fuels or fuel properties.

Infrared spectroscopy techniques offer an efficient and reliable means to estimate several fuel properties. These are not ASTM standardized methods, therefore they do not meet the current requirements of the PQAS. However, infrared spectroscopy methods could be used as screening tools. As ASTM continues to work on these types of methods, and as they become standardized, the Army should consider using them when appropriate.

VII. REFERENCES

1. Airmobile Aviation Fuel Laboratory (NSN 6640-00-902-9711), Operator's, Unit and Direct Support Maintenance Manual: TM 10-6640-216-13&P

Table 4. Fuel Properties Commonly Estimated By Ft-IR				
Fuel Property	Gasoline	Diesel	Kerosene	ASTM Procedure
Density @ 15ºC, g/mL	x	x	x	D 4052
Vapor Pressure, psi	x			D5191
Distillation, ºC	x	x	x	D 86
Research Octane Number (RON)	x			D 2699
Motor Octane Number (MON)	x			D 2700
(R +M)/2	x			calculated
Aromatic Hydrocarbons by FIA, vol%	x			D 1319
Olefinic Hydrocarbons by FIA, vol%	x			D 1319
Saturated Hydrocarbons by FIA, vol%	x			D 1319
Benzene, vol%	x			D 3606 or D 4420
Oxygenates, vol%	x			D 4815
Alcohols, vol%	x			D 4815
Total Oxygen, vol%	x			D 4815
Freeze Point, ºC			x	D 2386
Flash Point, ºC		x	x	D 93
Cloud Point, ºC		x	x	D 2500
Pour Point, ºC		x	x	D 97
Kinematic Viscosity @40ºC, cSt		x	x	D 445
Cetane Index		x	x	D 976 or D 4737
Hydrogen Content, mass%		x	x	D 5291
Carbon Content, mass%		x	x	D 5291
Net Heat of Combustion, MJ/Kg		x	x	D 240
Total Aromatics by SFC, mass%		x	x	D 5186

Table 5. Correlation Coefficients For Selected Fuel Properties (Measured During The Completed Pqas Pdrr Phase)	
Measured Property	Correlation Coefficient (r)
Distillation	
10 vol%	0.965
20 vol%	0.946
50 vol%	0.991
90 vol%	0.960
Final Boiling Point	0.916
Density	0.980
Cloud Point	0.550
Pour Point	0.558
Total Aromatics	0.991
Cetane Index	0.998
Knock Rating	0.744

Table 6. Standard Error of Prediction of Calibrations for Selected Diesel/Kerosene Properties			
Property	**Calibration Range Min.**	**Calibration Range Max.**	**Standard Error of Prediction**
Density	0.7883	0.8728	0.0024
Freeze Point	-59.5	2.3	5.2
Flash Point	38	85	7
Cloud Point	-60.5	-2.6	4.5
Pour Point	-75	-9	18
Viscosity at 40°C	1.15	3.92	0.17
Initial Boiling Point	139	217	11
10% Distillation	158	256	7
50% Distillation	194	297	6
90% Distillation	223	340	12
95% Distillation	230	358	14
End Point	249	372	12
Cetane Index, D976	37.4	59.8	1.0
Cetane Index, D4737	38.3	60.1	1.1
Hydrogen Content	12.29	14.19	0.10
Carbon Content	84.95	87.73	0.31
Carbon - Hydrogen Ratio	5.9213	7.0968	0.0513
Net Heat of Combustion, MJ/Kg	42.29	43.46	0.12
Total Aromatics	10.7	47.2	1.1